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(*S*)-Benzyl 2-[*N*-(5-chloropyridin-2-yl)-carbamoyl]pyrrolidine-1-carboxylate

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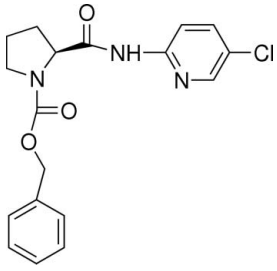
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.087; data-to-parameter ratio = 17.7.

In the title compound, $C_{18}H_{18}ClN_3O_3$, the pyrrolidine ring adopts an envelope conformation. The dihedral angle between the phenyl ring and the pyridine ring is 28.30 (7)°. The crystal packing is stabilized by N—H···O and C—H···O hydrogen bonding.

Related literature

For general background, see: Patel *et al.* (2002). For related structures, see He (2006); Kang *et al.* (2007).



Experimental

Crystal data

$C_{18}H_{18}ClN_3O_3$
M_r = 359.80
Orthorhombic, $P2_12_12_1$
a = 6.2419 (14) Å
b = 15.785 (4) Å
c = 17.574 (4) Å
V = 1731.5 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 (2) K
0.39 × 0.32 × 0.21 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: none
22340 measured reflections
3991 independent reflections
3602 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.088
S = 1.04
3991 reflections
226 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Absolute structure: Flack (1983), Friedel pairs 1686
Flack parameter: 0.02 (6)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O1^{i}	0.86	2.06	2.9148 (17)	172
C6—H6A···O3^{ii}	0.93	2.39	3.308 (2)	171

Symmetry codes: (i) $x+1, y, z$; (ii) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2339).

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supplementary materials

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(*S*)-Benzyl 2-[*N*-(5-chloropyridin-2-yl)carbamoyl]pyrrolidine-1-carboxylate

L. He

Comment

The title compound is an important inhibitor of peptidyl deformylase (Patel *et al.*, 2002). Its crystal structure is presented here.

The molecular structure is shown in Fig. 1. Bond lengths and angles are normal. The dihedral angle between the C6-benzene and O1/O2/N1/C8 planes is 50.56 (7)° and that between N3-pyridine and O3/N2/C12/C13 planes is 11.34 (11)°. The crystal packing is stabilized by N—H···O and C—H···O hydrogen bonding (Table 1).

Experimental

N-Carbobenzyloxy-*L*-proline (1.0 g, 4 mmol) and triethylamine (0.4 g, 4 mmol) were dissolved in THF (15 ml). To the solution was added dropwise ethylchloroformate (0.44 g, 4 mmol) at 273 K. After the solution was stirred for 30 min, 5-chloropyridin-2-amine (4 mmol) was added over a period of 10 min. The resulting solution was stirred at 273 K for 1 h and then stirred at room temperature for a further 12 h, and finaly refluxed for 3 h. After cooling down to room temperature, the solution was diluted with ethyl acetate. After filtration and removal of solvent under reduced pressure, the residue was purified through column chromatography on silica gel to give the title compound. Colourless single crystals were obtained by recrystallization from an ethanol solution.

Refinement

H atoms were placed at calculated positions with C–H = 0.93 or 0.97 Å and N—H = 0.86 Å, and refined using a riding model with U_{iso}(H) = 1.2U_{eq}(C,*N*).

Figures

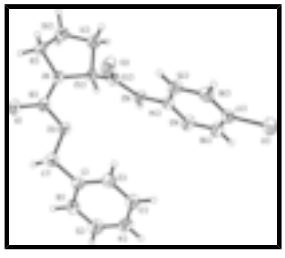


Fig. 1. The molecular structure of (I) with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

(I)

Crystal data

$C_{18}H_{18}ClN_3O_3$ F_{000} = 752

M_r = 359.80	D_x = 1.380 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 8860 reflections
a = 6.2419 (14) Å	θ = 1.7–27.5º
b = 15.785 (4) Å	μ = 0.24 mm^{-1}
c = 17.574 (4) Å	T = 293 (2) K
V = 1731.5 (7) Å^3	Block, colourless
Z = 4	0.39 × 0.32 × 0.21 mm

Data collection

Bruker APEXII area-detector diffractometer	3602 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.021
Monochromator: graphite	θ_{max} = 27.5º
T = 293(2) K	θ_{min} = 1.7º
ω scans	h = −8→8
Absorption correction: none	k = −20→20
22340 measured reflections	l = −22→22
3991 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.032	$w = 1/[\sigma^2(F_o^2) + (0.0474P)^2 + 0.1826P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.088	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.04	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
3991 reflections	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
226 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), Friedel pairs 1686
Secondary atom site location: difference Fourier map	Flack parameter: 0.02 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.95345 (10)	−0.20996 (3)	0.56412 (3)	0.07666 (17)
O1	−0.26937 (16)	0.14607 (7)	0.81673 (7)	0.0519 (3)
O2	0.06298 (16)	0.14500 (7)	0.76457 (6)	0.0501 (3)
O3	0.1628 (2)	−0.07143 (9)	0.80223 (8)	0.0693 (4)
N1	0.0084 (2)	0.07818 (8)	0.87283 (7)	0.0475 (3)
N2	0.4701 (2)	−0.00045 (8)	0.77641 (7)	0.0454 (3)
H2B	0.5368	0.0460	0.7861	0.054*
N3	0.7472 (2)	−0.02296 (8)	0.69420 (8)	0.0536 (3)
C1	0.1709 (3)	0.21399 (11)	0.65153 (8)	0.0496 (3)
C2	0.3125 (3)	0.15325 (12)	0.62553 (10)	0.0624 (4)
H2A	0.2895	0.0966	0.6374	0.075*
C3	0.4874 (3)	0.17575 (12)	0.58222 (10)	0.0653 (5)
H3A	0.5817	0.1342	0.5653	0.078*
C4	0.5231 (3)	0.25886 (13)	0.56400 (10)	0.0640 (5)
H4A	0.6412	0.2741	0.5348	0.077*
C5	0.3837 (3)	0.31895 (12)	0.58906 (12)	0.0688 (5)
H5A	0.4071	0.3755	0.5766	0.083*
C6	0.2077 (3)	0.29728 (11)	0.63282 (10)	0.0581 (4)
H6A	0.1142	0.3392	0.6496	0.070*
C7	−0.0186 (3)	0.19016 (14)	0.69944 (11)	0.0647 (5)
H7A	−0.0953	0.2405	0.7156	0.078*
H7B	−0.1160	0.1545	0.6707	0.078*
C8	−0.0816 (2)	0.12430 (9)	0.81787 (9)	0.0435 (3)
C9	−0.1015 (3)	0.05911 (12)	0.94413 (10)	0.0595 (4)
H9A	−0.1788	0.0059	0.9409	0.071*
H9B	−0.2007	0.1039	0.9578	0.071*
C10	0.0824 (3)	0.05351 (14)	1.00050 (11)	0.0692 (5)
H10A	0.1218	0.1092	1.0192	0.083*
H10B	0.0453	0.0177	1.0434	0.083*
C11	0.2603 (3)	0.01519 (13)	0.95454 (10)	0.0615 (4)
H11A	0.2470	−0.0460	0.9528	0.074*
H11B	0.3988	0.0297	0.9760	0.074*
C12	0.2343 (2)	0.05386 (10)	0.87433 (9)	0.0452 (3)
H12A	0.3268	0.1036	0.8682	0.054*
C13	0.2818 (2)	−0.01228 (9)	0.81333 (9)	0.0443 (3)
C14	0.5688 (2)	−0.05511 (9)	0.72421 (8)	0.0429 (3)
C15	0.4942 (3)	−0.13553 (10)	0.70621 (10)	0.0559 (4)
H15A	0.3676	−0.1560	0.7272	0.067*
C16	0.6120 (3)	−0.18417 (10)	0.65659 (10)	0.0591 (4)
H16A	0.5669	−0.2384	0.6434	0.071*
C17	0.7966 (3)	−0.15139 (10)	0.62707 (9)	0.0526 (4)
C18	0.8584 (3)	−0.07138 (12)	0.64670 (10)	0.0577 (4)
H18A	0.9840	−0.0498	0.6259	0.069*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0931 (4)	0.0654 (3)	0.0714 (3)	0.0266 (3)	0.0201 (3)	−0.0079 (2)
O1	0.0333 (5)	0.0546 (6)	0.0679 (7)	0.0003 (5)	0.0041 (5)	−0.0087 (5)
O2	0.0356 (5)	0.0637 (6)	0.0511 (6)	0.0016 (5)	0.0046 (4)	0.0090 (5)
O3	0.0523 (6)	0.0655 (7)	0.0901 (9)	−0.0205 (6)	0.0217 (7)	−0.0175 (7)
N1	0.0359 (6)	0.0534 (7)	0.0533 (7)	0.0013 (5)	0.0124 (5)	0.0040 (6)
N2	0.0392 (6)	0.0433 (6)	0.0536 (7)	−0.0051 (5)	0.0092 (5)	−0.0038 (5)
N3	0.0452 (7)	0.0544 (8)	0.0611 (8)	−0.0054 (6)	0.0140 (6)	−0.0098 (6)
C1	0.0451 (8)	0.0596 (9)	0.0440 (7)	0.0048 (7)	−0.0019 (6)	0.0045 (7)
C2	0.0672 (11)	0.0545 (9)	0.0655 (10)	0.0071 (8)	0.0099 (9)	0.0058 (8)
C3	0.0619 (11)	0.0719 (11)	0.0621 (10)	0.0156 (9)	0.0104 (9)	−0.0038 (9)
C4	0.0516 (10)	0.0830 (13)	0.0573 (10)	−0.0071 (9)	0.0055 (8)	0.0029 (9)
C5	0.0689 (12)	0.0586 (10)	0.0789 (12)	−0.0106 (9)	0.0054 (10)	0.0026 (9)
C6	0.0559 (10)	0.0561 (9)	0.0622 (9)	0.0067 (8)	0.0023 (8)	−0.0062 (8)
C7	0.0445 (8)	0.0875 (13)	0.0620 (10)	0.0099 (9)	0.0012 (8)	0.0202 (9)
C8	0.0350 (7)	0.0429 (7)	0.0527 (8)	−0.0051 (6)	0.0051 (6)	−0.0083 (6)
C9	0.0516 (9)	0.0617 (10)	0.0651 (10)	0.0003 (8)	0.0228 (8)	0.0061 (8)
C10	0.0761 (13)	0.0759 (12)	0.0555 (10)	0.0100 (10)	0.0166 (9)	0.0082 (9)
C11	0.0577 (10)	0.0761 (12)	0.0506 (9)	0.0106 (9)	0.0049 (8)	0.0018 (8)
C12	0.0351 (7)	0.0506 (8)	0.0498 (8)	0.0005 (6)	0.0073 (6)	0.0003 (6)
C13	0.0368 (7)	0.0462 (8)	0.0498 (8)	−0.0028 (6)	0.0057 (6)	0.0034 (6)
C14	0.0403 (7)	0.0455 (7)	0.0429 (7)	0.0008 (6)	0.0037 (6)	0.0011 (6)
C15	0.0595 (10)	0.0471 (8)	0.0611 (9)	−0.0075 (7)	0.0118 (8)	−0.0007 (7)
C16	0.0752 (12)	0.0412 (7)	0.0610 (10)	0.0023 (8)	0.0059 (9)	−0.0033 (7)
C17	0.0603 (10)	0.0505 (8)	0.0470 (8)	0.0160 (7)	0.0028 (7)	−0.0004 (7)
C18	0.0500 (9)	0.0618 (10)	0.0611 (10)	−0.0013 (8)	0.0144 (8)	−0.0059 (8)

Geometric parameters (Å, °)

Cl1—C17	1.7429 (16)	C5—H5A	0.9300
O1—C8	1.2215 (18)	C6—H6A	0.9300
O2—C8	1.3411 (18)	C7—H7A	0.9700
O2—C7	1.441 (2)	C7—H7B	0.9700
O3—C13	1.2091 (18)	C9—C10	1.519 (3)
N1—C8	1.334 (2)	C9—H9A	0.9700
N1—C9	1.4601 (19)	C9—H9B	0.9700
N1—C12	1.462 (2)	C10—C11	1.501 (3)
N2—C13	1.3552 (18)	C10—H10A	0.9700
N2—C14	1.4018 (18)	C10—H10B	0.9700
N2—H2B	0.8600	C11—C12	1.545 (2)
N3—C18	1.328 (2)	C11—H11A	0.9700
N3—C14	1.3326 (19)	C11—H11B	0.9700
C1—C6	1.374 (2)	C12—C13	1.526 (2)
C1—C2	1.382 (2)	C12—H12A	0.9800
C1—C7	1.500 (2)	C14—C15	1.389 (2)
C2—C3	1.377 (3)	C15—C16	1.375 (2)

C2—H2A	0.9300	C15—H15A	0.9300
C3—C4	1.369 (3)	C16—C17	1.365 (3)
C3—H3A	0.9300	C16—H16A	0.9300
C4—C5	1.360 (3)	C17—C18	1.365 (3)
C4—H4A	0.9300	C18—H18A	0.9300
C5—C6	1.384 (3)		
C8—O2—C7	115.93 (12)	C10—C9—H9B	111.3
C8—N1—C9	122.41 (13)	H9A—C9—H9B	109.2
C8—N1—C12	124.27 (12)	C11—C10—C9	103.41 (14)
C9—N1—C12	112.57 (13)	C11—C10—H10A	111.1
C13—N2—C14	127.55 (13)	C9—C10—H10A	111.1
C13—N2—H2B	116.2	C11—C10—H10B	111.1
C14—N2—H2B	116.2	C9—C10—H10B	111.1
C18—N3—C14	117.81 (14)	H10A—C10—H10B	109.0
C6—C1—C2	118.54 (16)	C10—C11—C12	104.72 (14)
C6—C1—C7	120.39 (16)	C10—C11—H11A	110.8
C2—C1—C7	121.07 (16)	C12—C11—H11A	110.8
C3—C2—C1	120.71 (17)	C10—C11—H11B	110.8
C3—C2—H2A	119.6	C12—C11—H11B	110.8
C1—C2—H2A	119.6	H11A—C11—H11B	108.9
C4—C3—C2	120.37 (17)	N1—C12—C13	110.77 (13)
C4—C3—H3A	119.8	N1—C12—C11	102.82 (12)
C2—C3—H3A	119.8	C13—C12—C11	110.52 (13)
C5—C4—C3	119.24 (18)	N1—C12—H12A	110.8
C5—C4—H4A	120.4	C13—C12—H12A	110.8
C3—C4—H4A	120.4	C11—C12—H12A	110.8
C4—C5—C6	121.01 (18)	O3—C13—N2	124.20 (14)
C4—C5—H5A	119.5	O3—C13—C12	121.48 (13)
C6—C5—H5A	119.5	N2—C13—C12	114.25 (13)
C1—C6—C5	120.13 (16)	N3—C14—C15	122.55 (14)
C1—C6—H6A	119.9	N3—C14—N2	113.06 (13)
C5—C6—H6A	119.9	C15—C14—N2	124.37 (14)
O2—C7—C1	106.91 (13)	C16—C15—C14	118.41 (16)
O2—C7—H7A	110.3	C16—C15—H15A	120.8
C1—C7—H7A	110.3	C14—C15—H15A	120.8
O2—C7—H7B	110.3	C17—C16—C15	118.72 (16)
C1—C7—H7B	110.3	C17—C16—H16A	120.6
H7A—C7—H7B	108.6	C15—C16—H16A	120.6
O1—C8—N1	124.73 (14)	C18—C17—C16	119.56 (15)
O1—C8—O2	124.45 (15)	C18—C17—Cl1	119.50 (14)
N1—C8—O2	110.81 (12)	C16—C17—Cl1	120.94 (13)
N1—C9—C10	102.49 (14)	N3—C18—C17	122.93 (16)
N1—C9—H9A	111.3	N3—C18—H18A	118.5
C10—C9—H9A	111.3	C17—C18—H18A	118.5
N1—C9—H9B	111.3		
C6—C1—C2—C3	0.4 (3)	C8—N1—C12—C11	171.05 (14)
C7—C1—C2—C3	−179.33 (16)	C9—N1—C12—C11	0.82 (18)
C1—C2—C3—C4	−0.3 (3)	C10—C11—C12—N1	−23.25 (18)

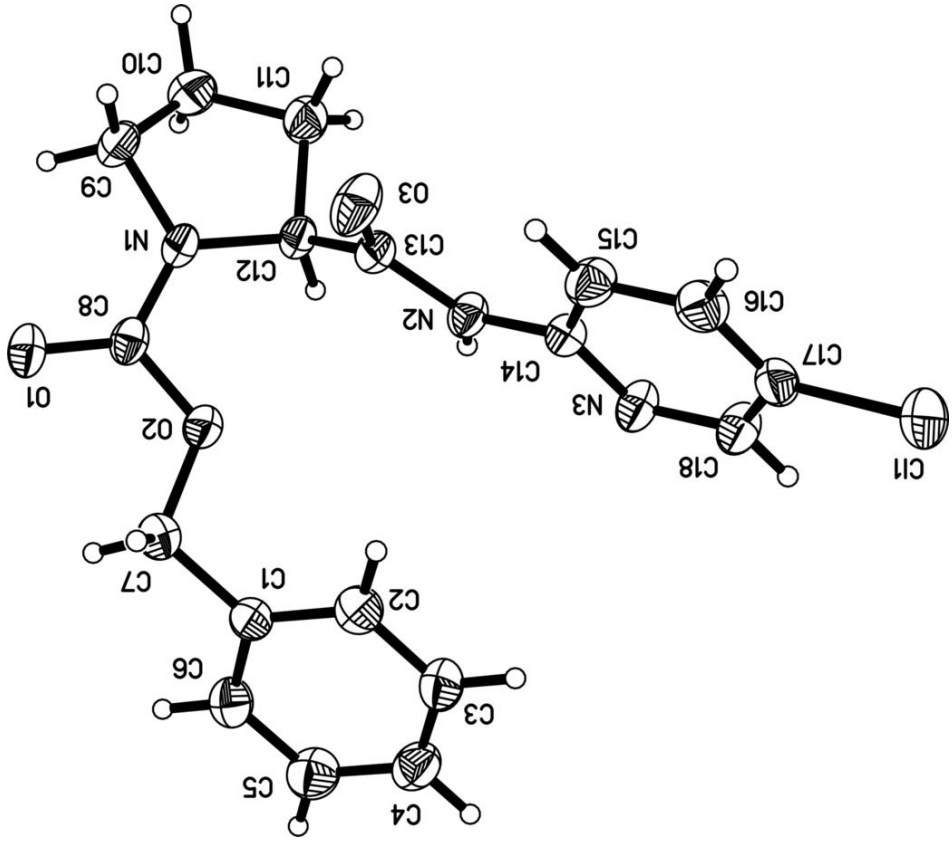
C2—C3—C4—C5	−0.1 (3)	C10—C11—C12—C13	−141.51 (15)
C3—C4—C5—C6	0.3 (3)	C14—N2—C13—O3	−5.0 (3)
C2—C1—C6—C5	−0.2 (3)	C14—N2—C13—C12	172.18 (13)
C7—C1—C6—C5	179.54 (17)	N1—C12—C13—O3	−43.5 (2)
C4—C5—C6—C1	−0.2 (3)	C11—C12—C13—O3	69.8 (2)
C8—O2—C7—C1	174.62 (14)	N1—C12—C13—N2	139.26 (13)
C6—C1—C7—O2	−123.60 (17)	C11—C12—C13—N2	−107.46 (15)
C2—C1—C7—O2	56.1 (2)	C18—N3—C14—C15	−1.6 (2)
C9—N1—C8—O1	−9.5 (2)	C18—N3—C14—N2	176.92 (15)
C12—N1—C8—O1	−178.78 (15)	C13—N2—C14—N3	176.11 (14)
C9—N1—C8—O2	169.35 (14)	C13—N2—C14—C15	−5.5 (2)
C12—N1—C8—O2	0.0 (2)	N3—C14—C15—C16	1.3 (3)
C7—O2—C8—O1	−5.2 (2)	N2—C14—C15—C16	−177.01 (15)
C7—O2—C8—N1	175.97 (14)	C14—C15—C16—C17	−0.1 (3)
C8—N1—C9—C10	−149.02 (15)	C15—C16—C17—C18	−0.7 (3)
C12—N1—C9—C10	21.42 (19)	C15—C16—C17—Cl1	179.81 (14)
N1—C9—C10—C11	−35.11 (19)	C14—N3—C18—C17	0.7 (3)
C9—C10—C11—C12	36.48 (19)	C16—C17—C18—N3	0.4 (3)
C8—N1—C12—C13	−70.87 (18)	Cl1—C17—C18—N3	179.94 (14)
C9—N1—C12—C13	118.90 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2B···O1^{i}	0.86	2.06	2.9148 (17)	172
C6—H6A···O3^{ii}	0.93	2.39	3.308 (2)	171

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*, *y*+1/2, −*z*+3/2.

Fig. 1


C10
C11
C9
O3
N1
C13
C15
C12
C8
N2
C16
C14
C17
O1
N3
O2
C18
Cl1
C7
C1
C2
C6
C3
C5
C4